Production of Polymer Test Particle Standards by Inkjet Printing

NIST researchers have explored the use of a novel inkjet printing approach to prepare polymer microspheres containing explosives and explosives simulants to be used as test standards for calibrating explosive detection portals. There is an immediate need for standard test particulate materials for calibrating or verifying trace explosives detection portals, a new technology being developed for rapid screening of people for high explosives to support homeland security.

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The preparation of realistic standard test materials for explosive portal systems requires fabrication of monodisperse particles of known size and compositions deposited onto an appropriate substrate in well characterized numbers. One intriguing method for generation of such test standards is the production of uniform polymer microspheres containing explosives or explosive simulant compounds of interest. Microspheres are attractive for this application because they are monodisperse and can be prepared in an appropriate size range for testing portal systems. Another advantage is a potential reduction in vapor pressure of the analyte compounds encapsulated in the polymer matrix which may extend the useful lifetime of the standards. With regard to safety, a simulant compound delivered in a bio-friendly polymer matrix, such as poly(DL-lactide/glycolide) 85:15 (PLGA), would provide a test material that is both safe with regard to human exposure and non-contaminating with regard to actual explosives being detected.

The "sphere jet" system is a drop on demand inkjet printer with a piezoelectric driven tip consisting of a micro capillary with a 50 µm orifice diameter. In the current implementation of the technique, the inkjet printhead is submerged under water and used to extrude a polymer - analyte solution using pressure assisted flow. A fluorescent dye is typically added to the solution to aid in visualization of the spheres. The waveform and frequency driving the piezoelectric crystal are adjusted to produce individual microspheres. The particle size is controlled by the polymer concentration, fluid feed rate and the applied frequency of the waveform. The stream of polymer microspheres form by an oil-water emulsion reaction, but the uniformity of the spherical particles is controlled by the inkjet printing mechanism. An image of an actual inkjet printer producing a stream of monodisperse spherical droplets is shown in Figure 1. A video camera with strobe illumination is used to monitor the quality of the spheres being jetted. The microspheres are captured and cured in a 500 mL beaker containing filtered, deionized water continuously stirred on a magnetic stir plate for several minutes to several hours. Any solvent that remains after sphere formation is allowed to evaporate.

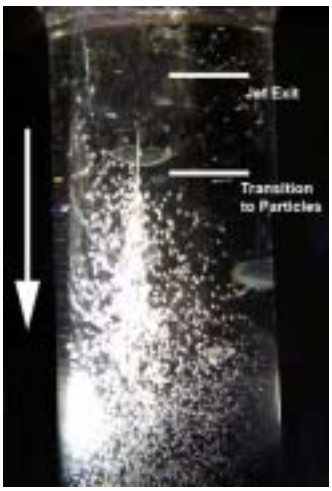
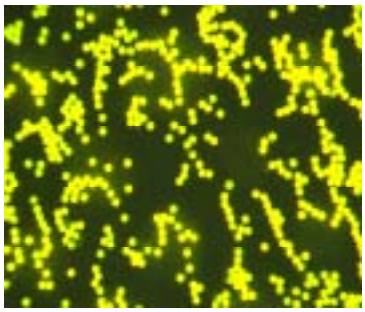
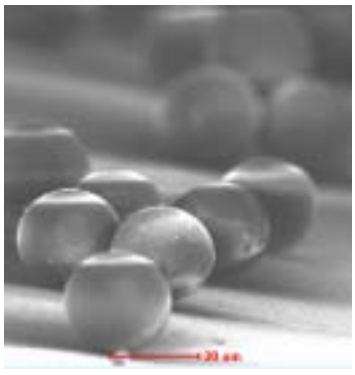


Figure 1. The sphere jet submerged in water producing PLGA spheres.





A fluorescence micrograph of PLGA spheres containing BHT and rhodamine B is presented in ***Figure 2*** (to the left) showing some of the PLGA doped spheres collected on a polycarbonate filter. The spheres are very uniform in size and shape. The scanning electron micrograph of PLGA – BHT spheres shown in ***Figure 3*** (to left) illustrates the uniformity of the spheres and absence of surface texture.

We have demonstrated the technique by producing monodisperse PLGA spheres containing the stimulant 2,6-Di-tert-butyl-4-methyl phenol (BHT) at two different concentrations. In addition we have incorporated two high explosive compounds into the PLGA sphere matrix. The final analyte concentration in the microspheres is measured by gas chromatography-mass spectrometry. All of the prepared microspheres have produced appropriate analyte signals in ion mobility spectrometry detection systems and the polymer matrix does not appear

to produce any additional ions that would interfere with detection.

We are pursuing research to improve production and to quantify the amount of analyte in the polymer microspheres and to determine the physical form and location of the analyte in the polymer matrix. More work is necessary to completely validate the approach and demonstrate its utility for making relevant test particles.

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